

10/747,668

PTO 07-5619

CC=JP DATE=19761222 KIND=A
PN=51149211

METHOD FOR PURIFYING ALIPHATIC AMINES
[Shibozoku Amin No Seiseiho]

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UNITED STATES PATENT AND TRADEMARK OFFICE
Washington, D.C. July 2007

Translated by: FLS, Inc.

PUBLICATION COUNTRY (19): JP
DOCUMENT NUMBER (11): 51149211
DOCUMENT KIND (12): A
PUBLICATION DATE (43): 19761222
PUBLICATION DATE (45):
APPLICATION NUMBER (21): 50073521
APPLICATION DATE (22): 19750617
INTERNATIONAL CLASSIFICATION (51): C07C 87/06; C07C 85/26
DOMESTIC CLASSIFICATION (52):
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TITLE (54): METHOD FOR PURIFYING ALIPHATIC
AMINES
FOREIGN TITLE [54A]: SHIBOZOKU AMIN NO SEISEIHO

1. Title of Invention

Method for Purifying Aliphatic Amines

2. Claim(s)

A method for purifying aliphatic amines characterized by adding, as the reducing agent, 0.01 to 2.0% by weight of an alkali metal salt of a borohydride with respect to a long-chain aliphatic amine using a natural fat or oil as the raw material, adding 0.1 to 5.0% by weight of an adsorbent having an alkali adsorption ability with respect to said amine next and at a proportion of at least 20 weight parts to 100 weight parts of said reducing agent.

3. Detailed Specifications

The present invention relates to a purification method in which the color tone and odor of long-chain aliphatic amines or and amine compound derivatives thereof manufactured by using a natural fat or oil as the raw material are improved.

In further detail, an improved purification method, wherein a borohydride compound is added to a long-chain aliphatic amine, reduced, and subsequently treated with an adsorbent; whereby the color tone and odor of this amine are improved, and further, the color tone and odor of an amine salt, quaternary ammonium salt, alkyl betaine, alkylene oxide adduct of an amine, and the like derived from this amine are improved, is provided in the present invention.

Aliphatic amines and their derivatives can be applied to diverse

* Numbers in the margin indicate pagination in the foreign text.

fields—from industrial products, such as fiber treatment agents, antistatic agents and emulsifiers, to cosmetics.

However, there are drawbacks because the odor of these aliphatic amines and their derivatives are extremely strong and the color tone is poor; hence, an improvement in these drawbacks has been demanded in the above-mentioned applications, and in particular, the cosmetics field.

A method in which an amine is purified with a borohydride compound to prevent an amine salt, quaternary ammonium salt or alkyl betaine derived therefrom from being colored is disclosed in Tokkai No. 49-95907 as a method of improvement thereof.

In this method, although the decoloring effect on amines is recognized definitely, no deodorizing effect is recognized at all. Moreover, boron oxide, which is a decomposition product of a borohydride compound, remains and this boron compound residue acts as an oxidization reaction catalyst when a derivative of the amine thereof, and in particular, a derivative obtained via a high-temperature reaction, such as an alkylene oxide /2 addition reaction or quaternary ammoniation reaction, undergoes a high-temperature reaction. The product degrades and the amount of odor constituent increases; hence, there is a drawback because the odor is extremely bad.

Oxidizable impurities, such as aldehydes and ketones, are contained in aliphatic amines using natural fats and oils as raw materials. They are odor sources or coloring precursors; hence, a long-chain aliphatic amine using a natural fat or oil as the raw material is more important in a method for solving coloring and odor problems than in the case of

an aliphatic amine using a petroleum product as the raw material.

Furthermore, an aliphatic amine increases the amount of the same oxidizable impurities causing oxidative degradation over time.

Consequently, the oxidizable impurities contained in the aliphatic amines must be removed, but it is necessary to remove the substances that will induce or promote an oxidation reaction.

As a result of painstaking research and investigations to solve these drawbacks, in view of the above problems, the inventors of the present invention discovered that there were remarkable advantages in improving color tone and odor by acting a borohydride alkali metal salt on a long-chain aliphatic amine using a natural oil or fat as the raw material and adsorbing it next with an adsorbent having alkali adsorption ability, which led them to completing the present invention.

That is, the present invention is a method for purifying aliphatic amines in which 0.01 to 2.0% by weight of an alkali metal salt of a borohydride with respect to a long-chain aliphatic amine are added as the reducing agent, a natural fat or oil is used as the raw material, 0.1 to 5.0% by weight of an adsorbent having an alkali adsorption ability with respect to said amine and at a proportion of at least 20 weight parts to 100 weight parts of said reducing agent are added next.

The amine which can be applied to the purification method of the present invention is an aliphatic amine using a natural fat or oil as a raw material, and in particular, an aliphatic amine having at least one alkyl group having eight or more carbons.

Especially effective amines include monoalkylamines,

aminopropylmonoalkylamines, monoalkyldiethylamines, monoalkyl-N,N-dihydroxyethylamines, dialkylamines and dialkylmonomethylamines.

Although a borohydride alkali metal salt selected from among sodium borohydride, potassium borohydride and lithium borohydride may be used for the borohydride compound of the present invention, on the contrary the advantages of the present invention cannot be obtained with hypophosphorous acid, hypophosphoric acid, sodium sulfite, hydrazines, etc.

The amount of the borohydride alkali metal salt used varies strictly depending on the type, attributes and treatment conditions of the amines. It ranges from 0.01 to 2.0% by weight (referred to as "%" hereinafter unless otherwise noted), and preferably, from 0.1 to 1.0% to the amine.

When it is less than the lower limit of 0.01%, there are few decoloring and deodorizing effects and the object of the present invention cannot be achieved.

And when it is greater than the upper limit of 2.0%, there is little improvement in the effect upon increasing the amount of use. When the amount used is extremely high, the borohydride compound of the reducing agent or its decomposition product is not removed sufficiently in the adsorption treatment performed next; hence, the treatment effects may be diminished.

An adsorbent having an alkali adsorption ability is used for the adsorbent used in the present invention, and in particular, silica, alumina, sodium oxide, magnesium oxide, and the like are preferred.

It is necessary to satisfy both of the conditions that the amount of this adsorbent used is 0.1 to 5.0%, and preferably, 0.5 to 2.0% with respect to the amine, and the ratio of 20 weight parts (simply referred to as "parts" hereinafter) or more to 100 parts of the above-mentioned reducing agent.

In this case, if the proportion of the adsorbent to the reducing agent is less than 20 parts of adsorbent to 100 parts of reducing agent, the synergistic effects of this adsorbent and reducing agent decline sharply.

Moreover, it is industrially uneconomical and unsuitable when the amount of the adsorbent used is less than 0.1% because the deodorizing effect is reduced by half, or when it exceeds 5.0% because an improvement in the treatment effects proportional to the increase in the amount added is not recognized.

Although the method for purifying an aliphatic amine of the present invention comprises both a reducing treatment step in which the borohydride alkali metal salt is added to the aforesaid amine first and an adsorption treatment step in which the adsorbent having an alkali adsorption /3 ability is added next, a high-temperature, long-term treatment greater than necessary should be avoided for the purpose of preventing the thermal degradation of the amine at the respective conditions of these treatments. As a consequence, the respective treatment conditions vary depending on the type of amine, but generally it is favorable to treat it at 30 to 100°C, and preferably, 50 to 80°C for 15 minutes to about 3 hours.

In a specific method of the present invention, the amine is accommodated in a container having a heating device and a stirring device, the borohydride

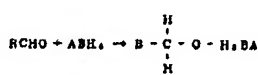
alkali metal salt is added thereto and reduced at the aforesaid treatment conditions in a nitrogen atmosphere, the aforesaid adsorbent having alkali adsorption ability is added next and stirred, normally for about 30 minutes to 1 hour and 30 minutes, cooled, subsequently filtered to separate and remove the borohydride alkali metal salt, adsorbent, and the like and obtain a purified amine.

In the aliphatic amine purified in the purification method of the present invention described in detail above, a more superior purified amine with improved color tone and odor can be obtained more easily and quickly than in a conventional method, as made clear from the practical examples and the like below.

Furthermore, the color tone and odor of the derivatives, such as amine salts and quaternary ammonium salts, derived from these aliphatic amines may be improved in the same way as well, and outstanding commodities, such as fiber treatment agents, antistatic agents, emulsifiers and beauty products, are obtained, unlike in the past.

Although the reasons the present invention provides such outstanding effects are not clear, they are inferred as follows.

That is, although large amounts of oxidizable impurities, such as aldehydes and ketones, are contained in the aliphatic amines using natural fats and oils, esters are produced by a reaction between these impurities and the borohydride alkali metal salt in accordance with the reaction formula shown next.

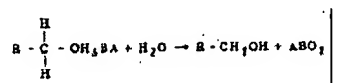


(providing A in the above formula is an alkali metal and B is an aliphatic

alkyl group).

When the adsorbent having alkali adsorption ability is used because the ester of the reaction product on the right side has an alkaline active group, the effects thereof are remarkable.

In addition, although this ester reacts with the moisture in the amine according to the following formula:



metaboric acid ions are generated and are dissolved in the amine as a source of coloring in the quaternizing reaction and the like of the amine, and the metaboric acid side reaction product ions (BO_2^-) also can be adsorbed and removed altogether.

That is, although just a borohydride compound is used as the reducing agent in the reducing treatment method used in a conventionally-known method for purifying an aliphatic amine, the borohydride compound in the method of the present invention adsorbs the impurities and has an effect for converting them intermediates that are more readily adhered adsorbed.

The present invention is described further next along with the test examples, practical examples, comparative examples, etc.

Test Examples

Comparison tests on the improvements in color tone and odor by the reduction-adsorption treatment of the method of the present invention and a conventionally-known adsorption-only treatment was performed by using distearylamine manufactured from a natural fat or oil raw material (97.3% pure amine) as the sample, selecting the reducing agent, sodium borohydride, and using "Kyoward 600S" (trade name, made by Kyowa Chemical

Industry Co., Ltd., 2 MgO·6 SiO₂·x H₂O) as the adsorbent.

These treatment effects were evaluated with an amine and a quaternary ammonium salt.

The color tone evaluation was performed using a Gardner-Hellige Colorimeter, and small numerals reflect outstanding decoloring effects.

Moreover, twenty specially trained test subjects were selected for the purpose of judging odors who provided their services at the workplace of an inventor of the present invention, to find how many (x) of the twenty subjects perceived the odor improving effects thereof according to the math expression: x subjects/20 subjects. As a consequence, in an example in which, e.g., 1/20 is an example of poor deodorizability, while 20/20 is an example of outstanding deodorization, showing that all twenty subjects could sense a deodorizing effect.

Table 1

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試料番号	1A 2A 3A 4A 5A 6A 7A 8A							
	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
脱色率 (%)	0.00	0.05	0.1	0.3	0.5	1.0	3.0	5.0
試験アミン	脱色	1 ⁺	1 ⁺	1 ⁺	1 ⁺	1 ⁺	1 ⁺	1 ⁺
結果の評価	脱色	5/20	11/20	15/20	17/20	18/20	20/20	20/20
試験四級	脱色	2	2	2	2	2	2	2
塩の評価	脱色	2/20	7/20	12/20	17/20	20/20	20/20	20/20
試料番号	1B	2B	3B	4B	5B	6B	7B	8B
脱色率 (%)	0.00	0.05	0.1	0.3	0.5	1.0	3.0	5.0
試験アミン	脱色	3	2 ⁻	2 ⁻	2 ⁻	2 ⁻	2 ⁻	2 ⁻
結果の評価	脱色	0/20	0/20	1/20	2/20	4/20	5/20	5/20
試験四級	脱色	3	3	3	3	3	3	3
塩の評価	脱色	0/20	0/20	0/20	3/20	5/20	6/20	7/20

Key:

Reduction-Adsorption Treatment Method (A)	Test No.			
	Treatment	Reducing Agent (%)		
		Adsorbent (%)		
	Evaluation of Results	Treatment evaluation of amine sample	Decoloring	
			Deodorization	
		Quaternary Salt Sample Treatment Evaluation	Decoloring	
Deodorization				
Adsorption Treatment Method Only (B)	Test No.			
	Treatment	Reducing Agent (%)		
		Adsorbent (%)		
	Evaluation of Results	Treatment evaluation of amine sample	Decoloring	
			Deodorization	
		Quaternary Salt Sample Treatment Evaluation	Decoloring	
Deodorization				

Notes: (1) The ■ symbol denotes a comparative example. (2) The adsorption treatment method (B) naturally is related to the comparative examples.

As is well understood from Table 1, it is easily clarified that the reduction-adsorption treatment method is outstanding. For example, in test no. 3A, it is $\frac{0.1}{0.5} \times 100 = 20\%$ since 0.5% of the reducing agent and 0.1% of the adsorbent are used. In the post-treatment evaluations, satisfying the conditions of the present invention also is outstanding.

It is well understood that the reduction-adsorption treatment method of the present invention is far more superior than the conventional adsorption treatment-only method.

Moreover, in test no. 1A of the reduction-adsorption treatment method in Table 1, no (0.00%) adsorbent was added at all and 0.5% reducing agent was added; hence, there is no effect by an adsorbent. At that time, the deodorizing effect of the quaternary ammonium salt is 2/20, and only the effect of the reducing agent can be seen by the "2" in the numerator thereof.

The treatment effects on this quaternary ammonium salt and the reduction-adsorption treatment method and adsorption treatment method

are illustrated as in the appended graph.

That is, curve A in the graph shows test nos. 1A to 8A in Table 1 and curve B shows 1B to 8B in Table 1 plotted as a curve; hence, when there is no adsorbent at all (point 1A), deodorizing is 2/20. Therefore, the deodorizing evaluation value x shown by the difference between at least curves comprising curve C using this as the starting point, point D and curve B and curve A indicates the synergistic effects of the reducing agent and adsorbent on curve A.

It is well understood that there is a sufficient effect when the amount of the adsorbent in the reduction-adsorption treatment method is about 0.3%, and that 1.0% or more approaches a constant value in terms of the effect thereof.

According to this practical example, the relationship between the constitution and advantages of the present invention may be clearly understood.

The practical examples and comparative examples are further described next.

Practical Examples and Comparative Examples

Comparative examples in which various amines are used as samples, and one of the reducing agent or the adsorbent used in the present invention is used and practical examples in which the treatment was performed by using both of these agents are shown below.

The methods for evaluating the color tone, odor, and the like in /5 each of these examples employed the same methods as in the aforementioned practical examples.

Moreover, a sample, which is a solid at room temperature, such as distearylamine, is coated on a 7 cm diameter filter paper, and moreover, contains a solvent, such as a quaternary ammonium salt, is evaluated after drying it for 10 minutes to remove the solvent.

In addition, a sample that is a liquid at room temperature, such as monoalkyldimethylamine, is evaluated after immersing a 7 cm diameter filter paper therein for 2 minutes and removing it.

Comparative Examples

A flask was charged with 500 g distearylmonomethylamine (98.6% pure amine) and heated to 75°C in a nitrogen atmosphere to dissolve the amine.

Next, 2.5 g sodium borohydride is added and subjected to a stirring and reducing treatment for 1 hour in a nitrogen atmosphere.

5.0 g of adsorbent were added thereto, treated for 0.5 hours at the same conditions as in the reducing treatment, a hot filtration (75°C) is performed using a Hyflo Super-Cel (trade name of filtration aid composed mainly of diatomaceous earth made by Johns-Manville Corp.) to remove the adsorbent, etc.

Quaternization is performed using this treated amine, and the color tone and odor are evaluated with amines and quaternary ammonium salt. The results obtained when the adsorption-treated amines and reduced amines with various adsorbents were used are compared and shown in Table 2.

Moreover, the treatment method of the adsorption-treated amine in this comparative example is performed under the same conditions as those of the adsorption treatment in the case of the above-mentioned practical examples. The adsorbent is removed by filtration to obtain an

adsorption-treated amine, and moreover, the treatment for reducing the reduced amine is the same as that in the practical examples.

Table 2

比較例番号	1	2	3	4
処理法	還元法	還元法	還元法	還元法
還元剤	—	S.B.H.	P.B.H.	S.B.H.
吸着剤	2MeO- 6810	—	—	—
試料74%	脱色 2"	1	2	2
の処理評価	脱色 3/20	2/20	1/20	1/20
試料90%	脱色 2	1	2	2
の処理評価	脱色 4/20	2/20	2/20	1/20

Key:

Comparative Example Test No.		Adsorption Method	Reducing Method	Reducing Method	Reducing Method
Treatment Method					
Reducing Agent					
Adsorbent					
Amine Sample Treatment Evaluation	Decoloring				
	Deodorization				
Quaternary Salt Sample Treatment Evaluation	Decoloring				
	Deodorization				

(Notes) S.B.H.: sodium borohydride; P.B.H.: potassium borohydride; ①: "Kyoward 600S" (trade name, made by Kyowa Chemical Industry Co., Ltd.)

Moreover, the methods for evaluating the decoloring and decolorization were performed using the same methods as in the practical examples (including the below-mentioned practical examples).

When the reducing agent and adsorbent are not used together as such, the effects are extremely low even when a definite % of each of these individual agents according to the present invention is added individually.

In addition, if the alkali of the borohydride is a sodium salt or potassium salt, the treatment effects thereof remain wholly unchanged.

Practical Example 1

Monostearyl dimethylamine (97.5% pure amine) manufactured by using

a natural fat or oil as the raw material was used as the sample.

Although the amount of the sodium borohydride used as the reducing agent was 0.5% respect to the amine, and moreover, the amount of the adsorbent used was 1.0%, the various types thereof changed.

Moreover, the adsorbents include "Kyoward #600S" (see Table 2), "Kyoward #700" ($\text{Al}_2\text{O}_3 \cdot 9\text{SiO}_2 \cdot x\text{H}_2\text{O}$), "Kyoward #400" ($\text{Na}_2\text{O}_3 \cdot \text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O}$), "Kyoward #500" ($6\text{MgO} \cdot \text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O}$) (all trade names of Kyowa Chemical Industry Co., Ltd.), etc.

These test results have been shown in Table 3.

Table 3

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試料	比較例	実例	実例	実例	実例	実例
番号	1	2	3	4	5	6
乳剤の濃度	—	—	S.B.H.	S.B.H.	S.B.H.	S.B.H.
還元剤	#600S	#700	#400	#500	#600S	#700
試料の濃度	3	3	3	3	3	3
20分間の脱色	1/20	2/20	15/20	14/20	10/20	20/20
試料の脱色	2	2	1	1	1	1
20分間の脱臭	3/20	4/20	14/20	16/20	20/20	20/20

Key:

Test No.		Comparative Example	Practical Example			
Treatment Method		Adsorption Method	Reduction-Adsorption Method (Method Of Practical Example)			
Reducing Agent						
Adsorbent						
Amine Sample Treatment Evaluation	Decoloring					
	Deodorization					
Quaternary Salt Sample Treatment Evaluation	Decoloring					
	Deodorization					

Substantially the same outstanding decoloring and deodorizing results were obtained even when the type of adsorbent was replaced, as shown in Table 3. It is clear that the results of the comparative examples are inferior.

Practical Example 2

This example was carried out with various amines using natural fats and oils as raw materials. The results in Table 4 were obtained next.

Moreover, sodium borohydride was used as the reducing agent, and the amount thereof used was 0.5% with respect to the amine, while 1.0% of the aforesaid "#600S," with respect to the amine, was used for the adsorbent.

Moreover, the names of the amines and the purities thereof are as shown in the table.

Table 4

試料番号	1	2	3	4	5
試料名	モノステアリン酸アミン	アミノエチルモノステアリン酸アミン	ジステアリン酸アミン	ジステアリン酸モノステアリン酸アミン	モノステアリン酸-N,N-ジヒドロエチルアミン
(純度)	(99.2%)	(99.0%)	(97.3%)	(98.8%)	(99.2%)
試料の色	1	3	1	1	3
脱色後の色	20/20	19/20	19/20	17/20	19/20
試料の臭気	1	3	1	1	2
脱臭後の臭気	20/20	20/20	20/20	18/20	17/20

Key:

Test No.					
Type of amine sample (Purity)		Monostearyl amine	Aminoethyl-monostearyl amine	Distearyl amine	Distearyl-monostearyl amine
Amine Sample Treatment Evaluation	Decoloring				
	Deodorization				
Quaternary Salt Sample Treatment Evaluation	Decoloring				
	Deodorization				

It is clear from Table 4 that the treatment in the method of the present invention is extremely outstanding.

Practical Example 3

The present invention was carried out by changing the type of the

various amine derivatives using natural oils and fats as raw materials.

Moreover, sodium borohydride is used as the reducing agent, while the amount thereof used is 0.5% with respect to the amine. The aforesaid "#600S" is used as the adsorbent, while the amount thereof used is 1.0% with respect to the amine.

Furthermore, comparative examples were described for comparing some samples.

Table 5

試験番号	比較例		実施例						
	1	2	3	4	5	6	7	8	9
製造法	還元法	鉄屑法	還元- 濃縮法	同左	同左	同左	同左	同左	同左
試料アミン の種類	モノスチ リルジ アルア ミン	同左	モノスチ リルア ミン	同左	モノスチ リルジ アルア ミン	同左	ジステア リルモノ アルア ミン	同左	モノスチ リルジ アルア ミン
(純度%)	(97.5)	(97.5)	(98.2)	(99.2)	(97.5)	(97.5)	(98.6)	(98.6)	(97.5)
溶媒 の種類	四級塩	同左	同左	アセト ン	アセト ン	アセト ン	四級塩	アセト ン	四級塩
結晶性	2	3	1	2	1+	2	1	1+	1
結晶性	3/20	4/20	19/20	20/20	18/20	20/20	20/20	18/20	20/20

Key :

Test No.		Comparative Example		Practical Example					
Treatment Method	Reducing method	Adsorption method	Reduction-Adsorption Method (Method of Practical Examples)	Same as at left	Same as at left	Same as at left	Same as at left	Same as at left	Same as at left
Type of Amine Sample (% purity)	Mono-stearyl dimethyl-amine	Same as at left	Mono-stearyl-amine	Same as at left	Mono-stearyl dimethylamine	Same as at left	Distearyl-monomethyl-amine	Same as at left	Mono-stearyl-dimethylamine
Type of Derivative	Quaternary salt	Same as at left	Same as at left	FO additive	Alkyl betaine	Fatty acid salt	Quaternary salt	Alkyl beta-ine	Hydro-chloride
Derivative Treatment Evaluation	Decoloring Deodorization								

It is evident from Table 5 that the practical examples of the present invention are outstanding.

Practical Example 4

A case in which the amount of adsorbent is changed is shown next.

In this case, sodium borohydride was used for the reducing agent, the amount thereof used was 1.0% with respect to the amine, while "Kyoward #600S" ($2 \text{ MgO} \cdot 6 \text{ SiO}_2 \cdot x \text{ H}_2\text{O}$) was used for the adsorbent.

In addition, the residual boron compound left in the treated amine was determined. A determination test using a coloring method with turmeric test paper was performed for the test method thereof by charging a reactor comprising a glass flask with 400 g of the respective treated amines,

and further, adding 100 g of warm water and stirring this for 1 hour at 75°C, letting it stand still to separate into layers, and separating out the bottom aqueous layer.

Moreover, the numerals shown by the items of this determination test indicated the magnification factors after separation of the aqueous layer, and in this case, at what magnification a turmeric coloring reaction was not observed when the boron aqueous solution was diluted.

The amine used in this practical example is distearylamine (97.3% amine).

Table 6 shows the evaluation results when the amount of adsorbent used is changed and the determination results of the boron compound using the turmeric test paper.

Table 6

実験番号	1	2	3	4	5	6	7
吸着剤使用量%	0.02 [■]	0.05 [■]	1.0	3.0	5.0	7.0 [■]	10.0 [■]
塩化アミン	脱色	1 ⁺	1	1	1	1	1
の 評 価	脱色	5/20	11/20	16/20	20/20	19/20	18/20
の 評 価	脱色	1	1	1	1	1	1
の 評 価	脱色	2/20	7/20	18/20	20/20	19/20	20/20
精査の定数	120	16	3	2	1.5	1.5	1.5

Key:

Test No.		
Amount of Adsorbent Used (%)		
Amine Sample Treatment Evaluation	Decoloring	
	Deodorization	
Quaternary Salt Sample Treatment Evaluation	Decoloring	
	Deodorization	
Boron Determination		

(Notes) The ■ symbol denotes a comparative example.

Practical Example 5

The results upon changing the amount of reducing agent are shown

next.

Moreover, sodium borohydride is used for the reducing agent, while "Kyoward #600S" is used for the adsorbent and the amount thereof used is 2.0% with respect to the amine. In addition, the amine used at this time is distearylamine (97.3% amine). The results of this test are as shown in Figure 7.

Table 7

試験番号	1	2	3	4	5	6	7
還元剤使用量	0.005	0.01	0.1	0.5	2.0	4.0	6.0
試料の脱色	2	1	1	1	1	1	1
の脱色	1/20	12/20	18/20	17/20	20/20	19/20	20/20
試料の脱色	2	1	1	1	1	1	1
の脱色	2/20	11/20	20/20	18/20	20/20	20/20	18/20

Key:

Test No.		
Amount of Reducing Agent Used (%)		
Amine Sample Treatment Evaluation	Decoloring	
	Deodorization	
Quaternary Salt Sample Treatment Evaluation	Decoloring	
	Deodorization	
Boron Determination		

(Notes) The ■ symbol denotes a comparative example.

It is evident from Table 7 that the purification method of the present invention is outstanding.

4. Brief Description of the Drawings

The drawing is a graph showing the synergistic effects of the present invention upon comparing the evaluations of the reduction-adsorption treatment method in a number of examples of embodiments of the present invention and a conventional adsorption treatment method using a deodorization evaluation value x typically using a quaternary amine in the test.